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**Methods for determination of hydrogen
chloride in flue gas**

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Foreword

This translation has been made based on the original Japanese Industrial Standard revised by the Minister of Economy, Trade and Industry, through deliberations at the Japanese Industrial Standards Committee as the result of proposal for revision of Japanese Industrial Standard submitted by Japan Environmental Measurement and Chemical Analysis Association (JEMCA)/Japanese Standards Association (JSA) with the draft being attached, based on the provision of Article 12 Clause 1 of the Industrial Standardization Law applicable to the case of revision by the provision of Article 14.

Consequently **JIS K 0107:2002** is replaced with this Standard.

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Methods for determination of hydrogen chloride in flue gas

1 Scope

This Japanese Industrial Standard specifies the methods for determination of hydrogen chloride in flue gas. In this Standard, the flue gas means the gas which is exhausted into flues, chimneys, ducts, etc. from such processes as burning and chemical reactions.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this Standard. For standards with the year indication, only the editions of the indicated year shall be applied and the revisions (including amendments) made thereafter shall not be applied. For those without the indication of the year, the most recent edition (including amendments) shall be applied.

JIS B 7984	<i>Continuous analyzer for hydrogen chloride in flue gas</i>
JIS K 0050	<i>General rules for chemical analysis</i>
JIS K 0055	<i>General rules for calibration method of gas analyzer</i>
JIS K 0095	<i>Methods for sampling of flue gas</i>
JIS K 0103	<i>Methods for determination of sulfur oxides in flue gas</i>
JIS K 0104	<i>Methods for determination of nitrogen oxides in flue gas</i>
JIS K 0106	<i>Methods for determination of chlorine in flue gas</i>
JIS K 0115	<i>General rules for molecular absorptiometric analysis</i>
JIS K 0122	<i>General rules for ion selective electrode method</i>
JIS K 0127	<i>General rules for ion chromatographic analysis</i>
JIS K 0557	<i>Water used for industrial water and wastewater analysis</i>
JIS K 8005	<i>Reference materials for volumetric analysis</i>
JIS K 8032	<i>Acetonitrile (Reagent)</i>
JIS K 8121	<i>Potassium chloride (Reagent)</i>
JIS K 8223	<i>Perchloric acid (Reagent)</i>
JIS K 8230	<i>Hydrogen peroxide (Reagent)</i>
JIS K 8295	<i>Glycerol (Reagent)</i>
JIS K 8355	<i>Acetic acid (Reagent)</i>
JIS K 8371	<i>Sodium acetate trihydrate (Reagent)</i>

JIS K 8541	<i>Nitric acid (Reagent)</i>
JIS K 8548	<i>Potassium nitrate (Reagent)</i>
JIS K 8550	<i>Silver nitrate (Reagent)</i>
JIS K 8576	<i>Sodium hydroxide (Reagent)</i>
JIS K 8622	<i>Sodium hydrogen carbonate (Reagent)</i>
JIS K 8625	<i>Sodium carbonate (Reagent)</i>
JIS K 8723	<i>Nitrobenzene (Reagent)</i>
JIS K 8830	<i>Uranine (Reagent)</i>
JIS K 8863	<i>Boric acid (Reagent)</i>
JIS K 8866	<i>Sodium tetraborate decahydrate (Reagent)</i>
JIS K 8891	<i>Methanol (Reagent)</i>
JIS K 8951	<i>Sulfuric acid (Reagent)</i>
JIS K 8962	<i>Potassium sulfate (Reagent)</i>
JIS K 8982	<i>Ammonium iron (III) sulfate 12-water (Reagent)</i>
JIS K 8987	<i>Sodium sulfate (Reagent)</i>
JIS K 9000	<i>Ammonium thiocyanate (Reagent)</i>
JIS K 9704	<i>2-Amino-2-hydroxymethyl-1,3-propanediol (Reagent)</i>
JIS K 9808	<i>2-Bis (2-hydroxyethyl) amino-2-hydroxymethyl-1,3-propanediol (Bis-Tris)</i>
JIS R 3503	<i>Glass apparatus for chemical analysis</i>
JIS Z 8808 : 1995	<i>Methods of measuring dust concentration in flue gas</i>

3 General matters

The general matters shall be as follows.

- General matters common to the chemical analysis shall be in accordance with **JIS K 0050**.
- General matters common to the sampling of flue gas shall be in accordance with **JIS K 0095**.
- General matters common to the ion chromatography shall be in accordance with **JIS K 0127**.
- General matters common to the ion selective electrode measuring method shall be in accordance with **JIS K 0122**.
- General matters common to the molecular absorptiometric analysis shall be in accordance with **JIS K 0115**.
- Water to be used in analysis shall be of A2 or A3 specified in clause 4 of **JIS K 0557**

or any water equivalent to these.

- g) Reagents to be used shall be of the highest grade or of an appropriate quality as specified in a relevant Japanese Industrial Standard, if applicable. If the reagent does not have a relevant specification in Japanese Industrial Standard, that of a quality adequate for the analysis shall be used.
- h) For the reference solution, the reference solution with traceability secured or that solution diluted to a definite concentration, and in addition, the solutions of which the preparation methods are specified for respective test items shall be used.
- i) Apparatus and instrument to be used shall be provided with the specified functions.
- j) When disposing flue gas, or absorbing solution of the flue gas used for the analysis of hydrogen chloride, it shall be done with utmost care.

NOTE 1 For the reagents with traceability secured, there are the reagents labelled with JCSS mark specified in the national measurement standard (Article 134 of the Measurement Act).

NOTE 2 The units of vol ppm and mg/m³ indicated in this Standard are the volume fraction and the mass concentration under the standard condition [273.15 K (0 °C), 101.32 kPa].

4 Classification and summary of analytical methods

The classification and summary of analytical methods shall be as given in table 1.

Table 1 Type and summary of analysis method

Type of analysis method	Summary of analysis method			Applicable condition
	Outline	Sampling	Determination range vol ppm (mg/m ³)	
Ion chromatography	After absorption of hydrogen chloride in sample gas into water, it is introduced into ion chromatograph and chromatogram thereof is recorded.	Absorbing bottle method Absorption solution : water Liquid amount : 25 ml ^{a)} × 2 bottles or 50 ml ^{b)} × 2 bottles Standard sampling volume : 20 L	0.4 to 7.9 ^{c)} (0.6 to 13) 6.3 to 160 (10 to 260)	According to 7.1.1
Silver nitrate titrimetry	After absorption of hydrogen chloride in sample gas into sodium hydroxide solution, it is made slight acidity, silver nitrate solution is added and the solution is titrated with ammonium thiocyanate solution.	Absorbing bottle method Absorption solution : sodium hydroxide solution of 0.1 mol/L Liquid amount : 50 ml ^{b)} × 2 bottles Standard sampling volume : 80 L	140 to 2 800 ^{d)} (230 to 4 600)	According to 7.2.1
Notes ^{a)} Liquid amount for absorption when using absorbing bottle (100 ml in capacity). ^{b)} Liquid amount for absorption when using absorbing bottle (250 ml in capacity). ^{c)} In the case of employing the sample solution for analysis prepared by diluting 50 ml of absorption solution through which the sample gas has been passed to 100 ml. The determination range shown in this column is obtained according to the standard sampling volume, the amount of sample solution for analysis and the optimum range of the working curve of the sample gas. When determining the concentration in this determination range or under, the concentrating column shall be used. For the concentration over the determination range, the sample solution for analysis shall be diluted to be within the determination range. ^{d)} In the case of employing the sample solution for analysis prepared by diluting 100 ml of absorption solution through which the sample gas has been passed to 250 ml. The determination range shown in this column is obtained according to the standard sampling volume, the amount of sample solution for analysis and the amount of titre.				

In addition, other than methods in table 1, the simultaneous analytical method of hydrogen chloride and sulfur oxides by ion chromatography is specified in Annex A, the simultaneous analysis method of hydrogen chloride, sulfur oxides and nitrogen oxides by ion chromatography is specified in Annex B and the ion selective electrode method is specified in Annex C. Also, the mercury (II) thiocyanate absorptiometry is specified in Annex D as reference.

5 Sampling method of sample gas

5.1 Sampling of sample gas

The sampling method of sample gas shall be as follows.

The sampling position of sample gas shall be selected from the point where representative sample gas can be sampled. The sample gas shall be taken at least twice without interval as far as possible, as a rule, at the same sampling position. Each of them shall be used for the analysis.

5.2 Reagents and preparation of reagent solutions

5.2.1 Reagents

The reagents shall be as follows.

- a) **Hydrogen peroxide** That specified in JIS K 8230.
- b) **Sodium hydroxide** That specified in JIS K 8576.

5.2.2 Preparation of reagent solutions

5.2.2.1 Absorption solution

The absorption solution shall be as follows.

- a) **For ion chromatography** Water specified in 3 f).
- b) **For silver nitrate titrimetry** Sodium hydroxide solution (0.1 mol/L). Weigh out 4.0 g of sodium hydroxide, and dissolve it in water to make total 1 L.

5.2.2.2 Sodium hydroxide solution (40 g/L)

This solution shall be put in a washing bottle. Dissolve 4 g of sodium hydroxide in water to make total 100 ml.

5.3 Implement and apparatus

5.3.1 Absorbing bottle

The absorbing bottle shall be as follows.

- a) **For ion chromatography** Two absorbing bottles (100 ml or 250 ml in capacity) connected as exemplified in figure 1 are used ¹⁾.
- b) **For silver nitrate titrimetry** Two absorbing bottles (250 ml in capacity) connected as exemplified in figure 1 are used ¹⁾.

Note ¹⁾ If the complete collection of hydrogen chloride is confirmed, it is permissible to use one absorbing bottle only.

5.3.2 Sampling apparatus of sample gas

The construction of the sampling apparatus is as exemplified in figure 2. The following conditions shall be satisfied.

- a) The sampling tube of sample gas (B) shall not be eroded by the corrosive gas in flue gas, and shall be made of the material which does not adsorb the sampling gas such as the stainless steel tube, borosilicate glass tube, quartz tube, tetrafluoroethylene resin tube.
- b) For preventing the dust contamination into the sample gas, the end or other suitable place of sampling tube of sample gas (B) shall be filled with appropriate filtering medium. The filtering medium by which the chemical reaction with the component in flue gas is not produced in material, such as silica wool, non-alkaline glass wool shall be used.
- c) For preventing the moisture condensation in the sample gas, the apparatus shall

be so constructed that the part from the sampling tube of sample gas (B) to the three-way cock for flow path switch (P_1) can be heated. The piping shall be as short as possible, and if there is a possibility of moisture condensation, the part between the sampling tube and the cock (P_1) shall be heated at about 120 °C.

When sulfur dioxides in flue gas are to be sampled at the same time, the said part shall be heated at about 160 °C.

- d) For the connection part heated from the sampling tube of sample gas (B) to the three-way cock for flow path switch (P_1), a stainless steel tube, borosilicate glass tube, fluoro rubber tube, silicone rubber tube, tetrafluoro ethylene resin tube and the like shall be used.
- e) The connection of each part in apparatus shall be assembled so as not to leak the gas.

5.4 Sampling procedure

The sampling procedures shall be as follows. The symbols of apparatus adopted here shall be as given in figure 2.

- a) Carry out either of the following.
 - 1) **For ion chromatography** Put 25 ml of absorption solution (water) of 5.2.2.1 a) into each of absorbing bottles (100 ml in capacity) (F_1 and F_2) ²⁾. In the case of using the absorbing bottles of 250 ml in capacity, put 50 ml of the absorption solution into each bottle.
 - 2) **For silver nitrate titrimetry** Put 50 ml of absorption solution of 5.2.2.1 b) specified for respective analytical methods into each of absorbing bottles (250 ml in capacity) (F_1 and F_2) ²⁾.
- b) Turn the three-way cocks for flow path switch (P_1 and P_2) to bypass side, start the suction pump (L), and replace inside air between the sampling tube of sample gas (B) and the cock (P_1) by the sample gas.

NOTE 1 The bypass (R) between P_1 and P_2 in figure 2 may be omitted, provided that the gas does not leak in a sampling apparatus and the flow rate can be adjusted to a definite amount.

NOTE 2 When it can be confirmed that the leak does not occur in the sampling apparatus by other technique, a mercury manometer (Q) in figure 2 may be omitted.

- c) After stopping the suction pump (L), close the three-way cocks for flow path switch (P_1 and P_2) to stop the flow of gas. Then, read the indication (V_1) of a gas meter (M) to the nearest 0.01 L.
- d) Start the suction pump (L), turn the three-way cocks for flow path switch and pass the sample gas through the absorbing bottles (F_1 and F_2) ²⁾. At this time, adjust a flow rate to about 1 L/min by adjusting the flow rate control cocks (K_1 and K_2) ³⁾. After sampling the sample gas of about 20 L ⁴⁾, close the three-way cocks for flow path switch (P_1 and P_2), stop the suction pump (L), and read the indication (V_2) of a

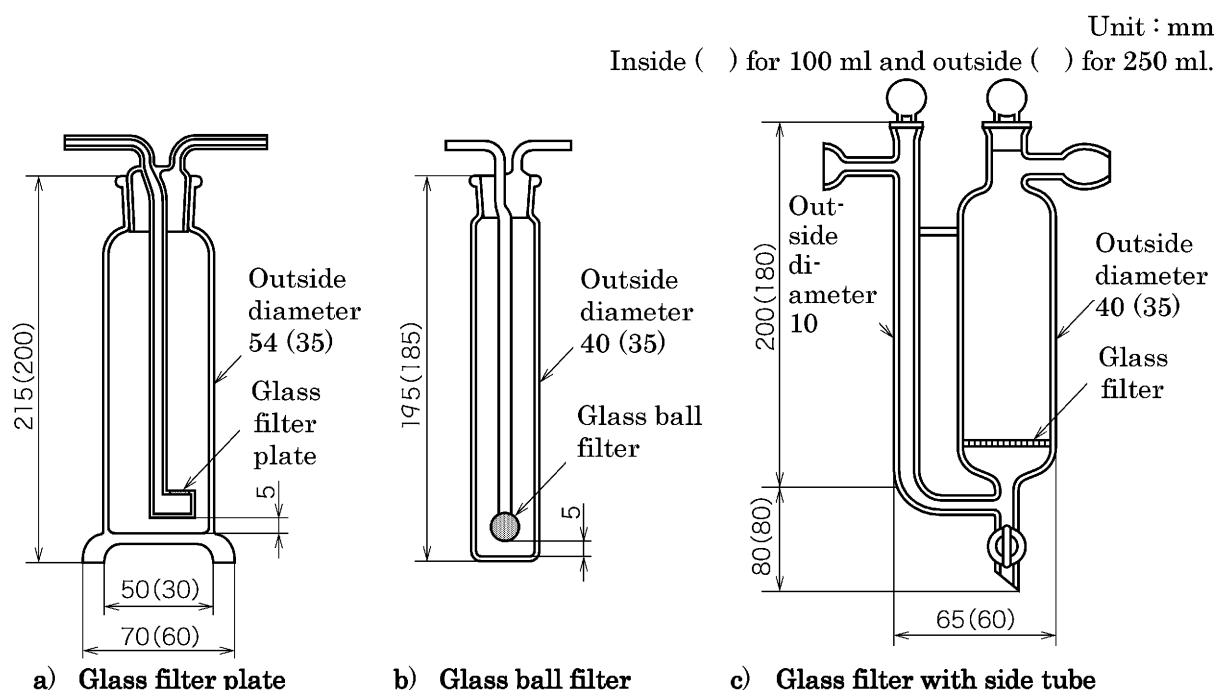
gas meter (M) to the nearest 0.01 L. At the same time, measure the temperature and the gauge pressure of the gas with the thermometer (N) and the manometer (O) of the gas meter (M). Also, measure the atmospheric pressure.

- e) If necessary, measure the moisture in the sample gas according to clause 6 of JIS Z 8808 : 1995.

Notes ²⁾ If there is a possibility that the absorption solution is warmed, place the absorbing bottles in a cooling tank. In this case, the absorbing bottle as shown in figure 1 a) should be used.

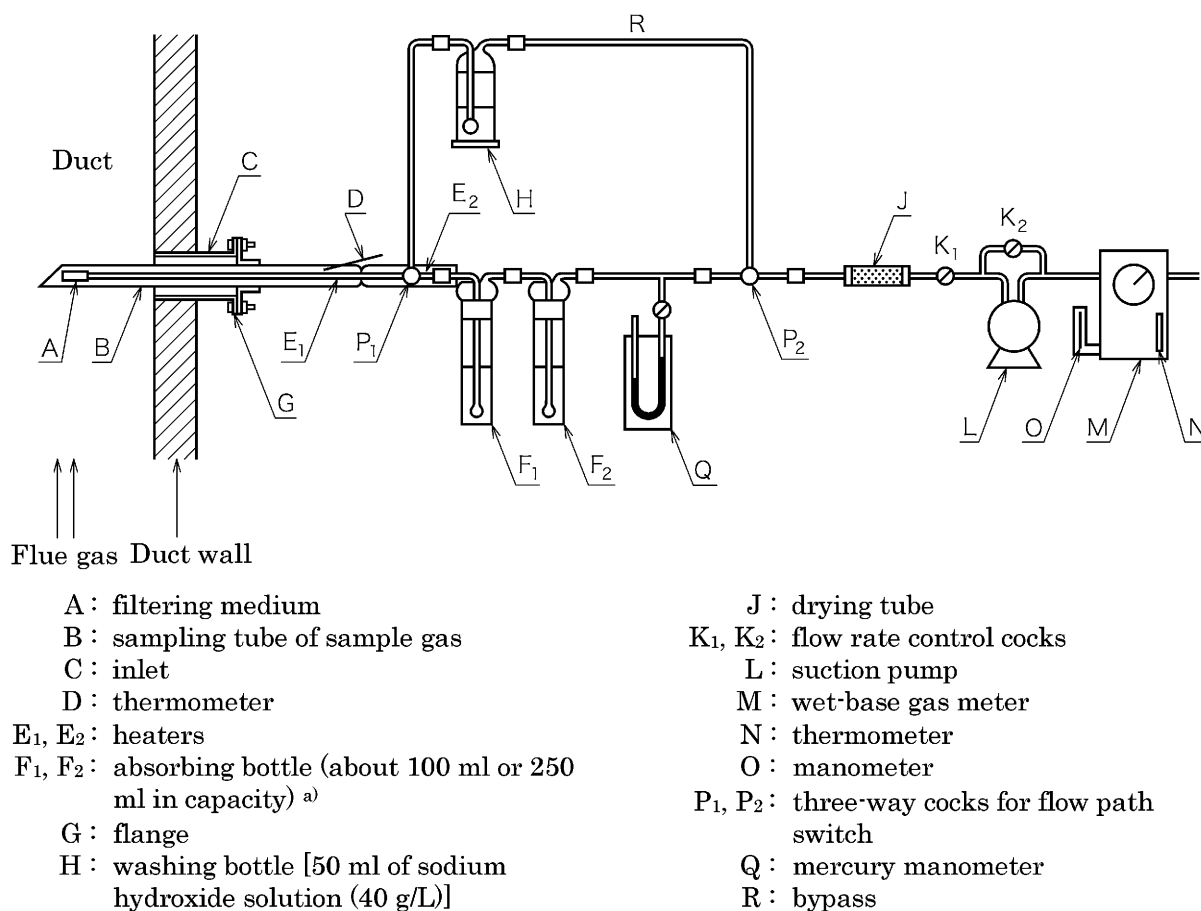
³⁾ When it has been known that hydrogen chloride will be completely absorbed in the absorption solution for the ion chromatography (when using an absorbing bottle of 250 ml in capacity) and the silver nitrate titrimetry, the flow rate may be increased up to 2 L/min.

⁴⁾ This amount may be suitably controlled according to the concentration of hydrogen chloride.



For the pore size of glass filter plate, glass ball filter and glass filter, the pore size symbol 1 or 2 specified in JIS R 3503 shall be used.

Figure 1 Example of absorbing bottle (100 ml and 250 ml)



Note ^{a)} If the complete collection of hydrogen chloride is confirmed, it is permissible to use one absorbing bottle only.

Figure 2 Example of sampling apparatus of sample gas

5.5 Sampling volume of sample gas

According to the following formulae, the sampling volume of sample gas under the standard conditions [273.15 K (0 °C), 101.32 kPa] shall be converted into the dry-base gas volume (V_{SD}) or the wet-base gas volume (V_{SW}).

a) For dry-base gas volume

1) When using a wet-base gas meter,

$$V_{SD} = V \times \frac{273.15}{273.15 + t} \times \frac{P_a + P_m - P_v}{101.32} + 22.41(a + b) \quad \dots\dots\dots(1)$$

2) When using a dry-base gas meter,

$$V_{SD} = V \times \frac{273.15}{273.15 + t} \times \frac{P_a + P_m}{101.32} + 22.41(a + b) \quad \dots\dots\dots(2)$$

b) For wet-base gas volume

1) When using a wet-base gas meter,

$$V_{SW} = V \times \frac{273.15}{273.15 + t} \times \frac{P_a + P_m - P_v}{101.32} + 22.41(a + b + c) \quad \dots\dots\dots(3)$$

2) When using a dry-base gas meter,

$$V_{\text{SW}} = V \times \frac{273.15}{273.15 + t} \times \frac{P_a + P_m}{101.32} + 22.41(a + b + c) \quad \text{..... (4)}$$

- where,
- V_{SD} : dry-base gas volume (L)
 - V_{SW} : wet-base gas volume (L)
 - V : gas volume measured by gas meter (L)
[$V_2 - V_1$ in procedures of **5.4 c)** and **d)**]
 - t : temperature at gas meter (°C)
 - P_a : atmospheric pressure (kPa)
 - P_m : gauge pressure at gas meter (kPa) ⁵⁾
 - P_v : saturated water vapour pressure at t °C (kPa) ⁶⁾
 - a : target gas collected in absorption solution (mol) ⁵⁾
 - b : gases other than target gas collected in absorption solution (mol) ⁵⁾
 - c : moisture amount obtained in clause **6** of **JIS Z 8808 : 1995** (mol) ⁵⁾
 - 273.15 : absolute temperature equivalent to 0 °C (K)
 - 101.32 : pressure corresponding to 1 atmospheric pressure (kPa)
 - 22.41 : volume of 1 mol gas under standard condition (L)

Notes ⁵⁾ In most cases, it can be neglected.

⁶⁾ About P_v , it is based on the conversion in table 2.

6 Preparation of sample solution for analysis

The preparation of sample solution for analysis shall be carried out according to any of the following.

a) For ion chromatography

- 1) After the procedures of **5.4**, transfer the solution in the absorbing bottles (F₁ and F₂) into a 300 ml beaker, wash the bottles, etc. with water, and join the washings to the said beaker.
- 2) When using absorbing bottles of 100 ml in capacity, transfer the solution in the beaker into a 100 ml volumetric flask by rinsing with water ⁷⁾, and add water up to the marked line. This is used for the sample solution for analysis.
- 3) When using absorbing bottles of 250 ml in capacity, transfer the solution in the beaker into a 250 ml volumetric flask by rinsing with water ⁷⁾, and add water up to the marked line. This is used for the sample solution for analysis.

Note ⁷⁾ If solid precipitate is found in the sample solution for analysis, it is recommended to remove it by filtering through the filter with 0.45 µm pore or under beforehand, because the separation column is blocked.

b) For silver nitrate titrimetry

- 1) After the procedures of 5.4, transfer the solution in the absorbing bottles (F₁ and F₂) into a 300 ml beaker, and wash the bottles, etc. with water.
- 2) Transfer the solution in the beaker into a 250 ml volumetric flask by rinsing with water, and add water up to the marked line. This is used for the sample solution for analysis.

7 Determination method

7.1 Ion chromatography

7.1.1 Condition of application

This method is affected by the coexistence of the reducing gas such as sulfides of high concentration in the sample gas, therefore, it is applicable for the case when the influence thereof can be neglected or eliminated.

7.1.2 Reagents and preparation of reagent solutions

7.1.2.1 Reagents

The reagents shall be as follows.

- a) **Sulfuric acid** That specified in JIS K 8951.
- b) **Sodium carbonate** That specified in JIS K 8625.
- c) **Sodium hydrogen carbonate** That specified in JIS K 8622.
- d) **Boric acid** That specified in JIS K 8863.
- e) **Sodium tetraborate decahydrate** That specified in JIS K 8866.
- f) **Sodium sulfate** That specified in JIS K 8987.
- g) **Potassium sulfate** That specified in JIS K 8962.
- h) **Potassium gluconate**
- i) ***p*-hydroxybenzoic acid**
- j) **Biochemical reagent-2-bis (2-hydroxyethyl) amino]-2-hydroxymethyl-1, 3-propanediol (bis-tris)** That specified in JIS K 9808.
- k) **Phthalic acid**
- l) **2-Amino-2-hydroxymethyl-1,3-propanediol** That specified in JIS K 9704.
- m) **Acetonitrile** That specified in JIS K 8032.
- n) **Glycerol** That specified in JIS K 8295.
- o) **Sodium chloride** Reference material for volumetric analysis specified in JIS K 8005.
- p) **Chloride-ion reference solution (Cl⁻ : 1 000 mg/L)**
- q) **Absorption solution** That prepared in 5.2.2.1 a).